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This article was submitted to the Boulder Symposium on Laser Optical Materials for High Power Lasers, Boulder, Colorado, October 1-3, 2001

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**December 12, 2001**

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This work was performed under the auspices of the U.S. Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

# Methods for mitigating growth of laser-initiated surface damage on fused silica optics at 351nm

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## ABSTRACT

We report a summary of the surface damage, growth mitigation effort at 351nm for polished fused silica optics. The objective was to experimentally validate selected methods that could be applied to pre-initiated or retrieved-from-service optics, to stop further damage growth. A specific goal was to obtain sufficient data and information on successful methods for fused silica optics to select a single approach for processing large aperture, fused-silica optics used in high-peak-power laser applications.

This paper includes the test results and the evaluation thereof, for several mitigation methods for fused silica surfaces. The mitigation methods tested in this study are wet chemical etching, cold plasma etching, CW-CO<sub>2</sub> laser processing, and micro-flame torch processing. We found that CW-CO<sub>2</sub> laser processing produces the most significant and consistent results to halt laser-induced surface damage growth on fused silica. We recorded successful mitigation of the growth of laser-induced surface damage sites as large as 0.5mm diameter, for 1000 shots at 351nm and fluences in the range of 8 to 13J/cm<sup>2</sup>, ~11ns pulse length. We obtained sufficient data for elimination of damage growth using CO<sub>2</sub> laser processing on sub-aperture representative optics, to proceed with application to large aperture (~40x40cm<sup>2</sup>) fused silica.

Keywords: laser damage, chemical etching, damage growth mitigation, plasma etching, CO<sub>2</sub> laser processing

## 1. INTRODUCTION

Surface damage initiated on polished fused silica surfaces during high-peak-power irradiation at 351nm encompasses only a fraction of the clear aperture area. However, studies have shown that the damage can grow with the number of shots at 351nm at laser fluences above ~5J/cm<sup>2</sup>, 11ns. [1]. A substantial increase in the useful lifetime of the optics can be achieved by stopping damage growth, thus mitigating obscuration caused by growing damage. Ideally this can be accomplished by eliminating the damages while they are small and returning the surface to its undamaged state. We explored several methods to mitigate the growth of UV-laser-induced damage on fused silica. Other studies at this Laboratory have focussed on elucidating the mechanisms for initiation [2] and growth [3] of surface damage on silica, whereas this effort seeks to identify and validate a leading method to successfully terminate the growth of such damage.

The following sections describe the experiments and results of damage growth tests for the four mitigation methods considered in this study; wet chemical etching, cold plasma etching, CW-CO<sub>2</sub> laser processing, and

micro-flame torch processing. The results show that CO<sub>2</sub> laser processing is consistent in mitigating damage and successfully stopping damage growth.

## 2. EXPERIMENTAL

In our experimental program, we tested both the intrinsic growth behavior of the mitigation pits and the growth of mitigated laser-initiated damage pits. It was necessary to determine that the pits formed by the mitigation methods themselves did not cause damage or grow with repeated illumination. In fact, this was the case for those methods that tended to contaminate the surface of the fused silica. All of the experiments used the same sample type; Corning 7980 fused silica, 50mm diameter, 10mm thick, and polished by SESO. For testing the pits produced by the mitigation methods on bare surface, the pits were arranged in an array of 6-9 spots spaced by 10mm. For the laser-initiated pits, we chose a common set of experimental conditions to place an array of 4-9 nominally uniform, equally spaced damage pits on the output (exit) surface of fused silica samples. Each laser-initiated damage site was produced by a single Gaussian-shaped pulse from a frequency-tripled Nd:YAG laser at 355nm, with a 0.9mm beam diameter, an average fluence of  $\sim 45\text{J/cm}^2$  and a pulse length  $\sim 7.5\text{ns}$ .

The typical surface damage produced by a single pulse at  $\sim 45\text{J/cm}^2$  consists of a cluster of 3 to 15 pits within about 0.3mm diameter; each pit in the group has a diameter in the range from 0.01-0.05mm (see figure 7). Although such surface damage is heavier than what is expected for typical damage on NIF optics, it was used as a worst-case condition for proof-of-principle mitigation testing. Thus, a successful demonstration of methods to mitigate the growth of such sites gives confidence to mitigate NIF related damage.

The growth tests were all carried out in a vacuum chamber operating at  $\sim 10^{-5}$  torr, in the slab laser facility at LLNL [1]. The sites were tested for growth using the frequency-tripled Nd-glass laser output, producing a 4mm x 6mm flat-top beam at 351 nm, with  $\sim 1\text{ns}$  pulse width at  $6\text{--}12\text{ J/cm}^2$ . The damage sites are illuminated by the laser at a rate of 1 pulse per 2 seconds. If the tested site grows, the growth rate is determined by measuring the occluded area as a function of the number of laser shots on a site. It is known from other LLNL work that the typical damage area grows at an exponential rate with the number of laser shots above a threshold  $3\omega$  fluence of about  $5\text{J/cm}^2$  [1]. In some cases, mitigated damage sites had a higher threshold for growth or the growth rate was not typical. Such results were duly noted, but the growth behavior itself was not explored since the primary objective was to study the efficacy of the mitigation methods.

## 3. WET-CHEMICAL ETCHING EXPERIMENTS AND RESULTS

Chemical etching with a hydrofluoric acid solution is a common method for dissolving silica and it is an accepted way to remove damage-affected material on silica surfaces. Ideally, this process will return the damage area to the state of the undamaged bulk material. This method was explored in previous work at LLNL to determine if mild etching (i.e., to a depth of  $\sim 600\text{nm}$ ) would remove precursors to laser-initiated damage [4]. The results showed a minor improvement over non-etched surfaces. Also, in previous work at LLNL, mild chemical etching was studied as a possible means to raise the growth threshold of UV-laser-induced damage [5]. The prior work used 2% HF solution to etch up to about 2 micrometers depth from the damage pits. The etched samples were damage growth tested in the slab laser facility at 351nm,  $5\text{--}6\text{ J/cm}^2$  ( $\sim 1\text{ns}$ ). The results showed a slight increase of the growth threshold for fused silica.

Since some change in the growth threshold was observed in the earlier studies, we decided to explore deeper etching for growth mitigation. We used 2% HF solutions to etch the entire surface of some damaged silica samples by dipping or we applied the solution directly to the damage pit using a fountain design [6], in other cases. Etch depths ranged from  $0.5\mu\text{m}$  to  $20\mu\text{m}$ . Damage growth tests were performed at 351nm ( $\sim 1\text{ns}$ ). A summary of the results of mitigation by etching at different etch depths is shown in Table 1.

Our measurements show that damage sites that have been etched to depths greater than about 9  $\mu\text{m}$  have about a 40% chance for zero growth with 1000 shots at fluences of 6.8-9.4  $\text{J}/\text{cm}^2$  and  $\sim 1\text{ns}$ . For the etched sites that grow in this fluence range, the growth rates are consistent with those for un-etched sites. Contamination of the sample surfaces was observed for both the dipped and fountain etched cases, which could affect the variable results. Figure 1 shows the growth coefficient vs. fluence data for samples that were etched to depths of 10 $\mu\text{m}$  and 20 $\mu\text{m}$ , as they compare to the statistical range of data for un-etched damage sites. These results are encouraging for possible mitigation of surface damage. More data is needed for smaller damage sites to determine if the statistics for complete mitigation of growth improve for such sites.

Table 1. Tabulated results for polished fused silica, surface damage growth tests at 351nm, 6.8-9.4 $\text{J}/\text{cm}^2$ ,  $\sim 1\text{ns}$ , of sites treated with a 2% HF solution etch.

Sample	Site	Etch depth( $\mu\text{m}$ )	Test fluence ( $\text{J}/\text{cm}^2$ )	Shots to cause growth
SC40021	G	10	7.8	1000+
	D	10	6.8	1000+
	I	10	8.9	1000+
	F	10	9.4	40
	H	10	8.4	1000+
	E	10	8.4	1000+
SC40023	C	0.5	7.8	3
	A	1	7.5	1
	I	3	8.4	2
	G	5	8.1	3
	E	10	8.1	1
SC40026	A	10	8.2	24
	B	10	8.1	17
	C	10	8.1	830
	D	10	7.8	1000+
	E	10	8.5	19
	F	10	8.4	23
SC40027	F	20	8.4	10
	E	20	8.2	1000+
	H	20	8.5	3
	G	20	7.4	1000+

#### 4. PLASMA-ETCHING EXPERIMENTS AND RESULTS

A radio-frequency (RF) plasma, spiked with fluorine-containing molecules, is commonly used to etch silica for integrated circuit applications. Fluorine atoms generated in the plasma, chemically attack the silicon-oxygen bonds of silica, forming a gas,  $\text{SiF}_4$ , thus effectively etching the material. With this approach to mitigation in mind, two types of plasma torches were investigated for damage growth mitigation. One type was a miniature version of a RF argon plasma torch (micro-plasma), fed by carbon tetrafluoride ( $\text{CF}_4$ ) gas, which etched small sites on fused silica. Figure 2 is a photo of the micro-plasma torch. The plasma diameter is about 1.5mm at the tip. When the torch is applied directly to the surface of the silica, it produces a  $\sim 2\text{mm}$  diameter pit; the pit depth ( $\sim 1\text{-}5\mu\text{m}$ ) depends on the exposure time. A typical pit profile is shown in Fig. 3. The second type of torch was a microwave plasma torch (nitrogen gas) also fed by  $\text{CF}_4$  gas, to produce fluorine atoms for reactive-atom plasma

processing (RAPP). The large RAPP torch shown in Fig.4, etches silica at a fast rate ( $\sim 0.2\mu\text{m}/\text{min}$ ) over a diameter of 10mm. The depth ( $\sim 10\text{-}100\mu\text{m}$ ) depends on the exposure time.

The initial work with the micro-plasma torch exposed several problems; 1) the etch rate is very slow ( $\sim 2\mu\text{m}/\text{hr}$ ), 2) the etching does not rapidly smooth the damage pit, and 3) the plasma produces contamination residue in the treated area. Evidence for 2) is seen in the profile shown in Fig. 3, where the original damage pit has receded into the substrate without smoothing as the etching takes place. Apparently, etching by the micro-torch is not isotropic as is typically the case for chemical solution etching. The initial experiments to apply the micro-plasma torch to etch laser-damage sites resulted in the damage receding into the site rather than being smoothed. These sites were not tested for growth because there was not a significant change in the damage structure after etching.

One sample was prepared for growth testing micro-plasma-produced pits on bare silica. All the pits were shallow ( $< 3\mu\text{m}$ ) due to low etch rates, and wide ( $\sim 1.8\text{mm}$ ). Microscopy of the pits before the tests disclosed some deposits located within each pit (see Fig.5). Analysis of the deposits showed that they contained carbon, which is believed to be residue from dissociation of the  $\text{CF}_4$  in the plasma. The laser fluence range for testing the growth at 351nm was  $6\text{-}8\text{J}/\text{cm}^2$ . All of the tested sites that had contamination developed a ‘stain’ within the first 20 shots, as they were illuminated at 351nm. These stains spread in the next 20-50 laser shots, to totally cover the original plasma-produced pit surface. The stains appeared to originate generally at or near visible contaminants within the pits.

Another sample was prepared for growth testing pits produced by the RAPP torch. Each site was about 10mm diameter, and the depths ranged from 20 –  $50\mu\text{m}$ . Microscopy disclosed deposits located within 3 of the 4 pits. When tested for growth, one site developed a ‘stain’ that grew, similar to the micro-plasma torch case. Two of the sites having visible contamination developed catastrophic growth (i.e., rapidly expanding damage) within 17 shots. The fourth site, a ‘clean’ site, had no visible change after 26 shots.

The results of tests for both of the plasma torch processes show that contamination in the mitigation pit tended to be unstable at the test fluences. These processes need additional development using non-carbon, fluorine containing compounds. Also, development is needed to speed the slow etch rate of the micro-plasma torch. Due to the failure of the bare mitigation pits to survive laser illumination without contamination induced growth, and because of the success with alternative methods, we did not proceed to test mitigated damage sites by either of the plasma torch methods.

## 5. $\text{CO}_2$ LASER EXPERIMENTS AND RESULTS

$\text{CO}_2$  lasers have been used successfully by others for reducing damage initiation on fused silica at 1054nm [7,8] but we were not aware of any attempts to use  $\text{CO}_2$  laser heating to treat existing damage on fused silica. We explored the use of  $\text{CO}_2$  lasers for mitigating 351nm laser-induced damage growth on fused silica. Preliminary experiments were done using an industrial size  $\text{CO}_2$  laser (Rofin-Sinar RS-1000, 1KW CW), primarily designed for cutting and welding. This laser was operated at reduced power ( $< 100\text{W}$  CW), with a focussed, Gaussian-shaped,  $1/e^2$  beam diameter of  $\sim 5\text{mm}$ , for one-second duration. The laser beam locally melted and evaporated the silica surface, typically producing smooth, Gaussian-shaped pits (see Fig.6). The pit depth ( $\sim 4\text{-}30\mu\text{m}$ ) depended on both laser power and de-focus.

The first damage tests at 351nm and  $\sim 11\text{ns}$  pulse width, showed that the  $\text{CO}_2$  laser-induced pits on undamaged silica, did not damage at fluences up to  $\sim 8\text{J}/\text{cm}^2$ . An extensive set of mitigation experiments was done for  $\text{CO}_2$  laser-treated, UV-laser-induced damage pits. Figure 6 shows examples of the UV-laser-induced damage and the pit produced after a one-second exposure of the  $\text{CO}_2$  laser at 50W. All of the  $\text{CO}_2$  laser-treated sites which were tested at 351nm,  $\sim 11\text{ns}$ , in the fluence range from  $\sim 6.7\text{-}12\text{J}/\text{cm}^2$ , survived 1000 shots. An example of the typical results for mitigating surface damage on fused silica is shown in Fig. 7.

After the initial successes with the CO<sub>2</sub> laser processing, three issues were identified for further consideration; 1) what is the mechanism for removal of material and what parameters determine the pit size and shape, 2) what is the affect on the wavefront propagation by the pit geometry and, 3) would simple thermal annealing by the CO<sub>2</sub> laser be sufficient to mitigate growth? All of these issues were addressed by modeling efforts and an experiment was also done to address the thermal annealing question.

A summary of the tests that were done for CO<sub>2</sub> mitigation is shown in Table 2. One site (SC40031-B) that was shot multiple times during initiation, which had very deep cracks associated with it, grew immediately. Four other sites on sample SC40031 were treated with laser conditions designed to test thermal annealing; all grew immediately when tested at 11-12J/cm<sup>2</sup>. All of the other 16 sites tested in the range of 6.7 to 12J/cm<sup>2</sup>, did not grow in 1000 or more shots, exhibiting complete mitigation of damage growth.

Table 2. Tabulated results for growth tests at 351nm, ~11ns, of damage sites treated with a CW-CO<sub>2</sub> laser.

Sample	Site	CO <sub>2</sub> treatment	# Shots @ Test fluence (J/cm <sup>2</sup> )	Comments
SC40036	A	50W, 1sec	1000 @ ~6.7	no growth
	B	50W, 1sec	1000 @ ~6.7	no growth
	C	none	35 @ ~6.7	control, normal growth
	D	50W, 1sec+μplasma	1000 @ ~6.7	darken, no growth
SC40037	A	37.5W, 1sec	1000 @ ~8.0	no growth
	B	37.5W, 1sec	1000 @ ~8.0	no growth
	C	37.5W, 1sec	1000 @ ~8.0	no growth
SC40029	A	37.5W, 1sec+μplasma	400 @ ~8.3	no growth
			1000 @ ~12.0	darken, no growth
	B	37.5W, 1sec	1000 @ ~8.3	no growth
	C	37.5W, 1sec+μplasma	1000 @ ~8.3	no growth
	D	none	20 @ ~8.3	control, normal growth
	E	37.5W, 1sec	1000 @ ~12.0	no growth
	G	37.5W, 1sec+μplasma	1000 @ ~8.3	darken, no growth
	H	37.5W, 1sec	1000 @ ~8.3	no growth
	I	37.5W, 1sec+μplasm	1000 @ ~8.3	darken, no growth
SC40031	A	27.5W, 1sec	1000 @ ~12.0	no growth
	B	27.5W, 1sec	7 @ ~12.0	xtra heavy damage
	C	27.5W, 1sec	1000 @ ~11.0	no growth
	D	27.5W, 1sec	1000 @ ~12.0	no growth
	E	17.7W, 60sec	4 @ ~12.0	grew on first shot
	F	none	6 @ ~12.0	control, grew first shot
	G	17.5W, 60sec	11 @ ~11.0	grew first shot
	H	17.5W, 60sec	5 @ ~12.0	grew first shot
	I	17.5W, 60sec	10 @ ~10.0	grew first shot

These results led us to conclude that CO<sub>2</sub> laser treatment can stop the growth of large (~0.3mm dia.) damage on polished fused silica surfaces at 351nm and ~11ns pulse length. It remains to be proven that mitigation works at shorter pulse lengths (eg., 3ns) and comparable frequencies, and that the mitigated sites do not re-initiate damage. The CO<sub>2</sub> laser process is not complex and it is amenable to rapid processing of a large number of sites on a given optic. It may also be possible to achieve growth mitigation by treating the entire optic surface, rather than point-

by-point; this would eliminate the difficult, tedious task to locate specific damage sites. On the other hand, maintaining surface figure for whole-surface treatment is clearly an issue.

Additional details of the experiments to test the CO<sub>2</sub> laser treatment for mitigation are published elsewhere in this Proceedings [9].

## 6. MICRO-FLAME TORCH EXPERIMENTS AND RESULTS

A hydrogen flame torch is another method to locally anneal a small damage site on fused silica. A miniature version of such a torch was developed and tested. The torch was fed by CF<sub>4</sub> to produce a hot flame with fluorine atoms that etch the silica surface. A schematic of the micro-flame torch system is shown in Fig.8. This torch represents an improvement over the RF plasma-torch for mitigation because it provides a combination of sufficient heat to soften the silica and fluorine atoms to etch the silica. It operates as an atomization source, wherein the hot flame dissociates the CF<sub>4</sub> molecules, producing F atoms, which attack the silica. The flame temperature can be varied to control the degree of dissociation. The torch produces a smooth, Gaussian shaped pit, approximately 1.5mm dia. and ~2 to 4μm deep, with no apparent lip. This torch was applied to treat 3 sites on a fused silica substrate.

A single sample was tested for the degree of mitigation at 351nm and ~11ns. The sample had 4 large damage sites (produced at ~45J/cm<sup>2</sup>, 355nm, 7.5ns), three of which were treated by the flame torch; one control site was not treated. One of the treated sites was totally mitigated against growth, i.e., it survived 1000 shots at 8J/cm<sup>2</sup>. Another one of the treated sites survived 200 shots at each of the test fluences, 8, 12 and 14J/cm<sup>2</sup>, but other surface damage appeared near the test site, and grew. The third treated site, which was not completely removed by the flame torch process, grew after 20 shots at 8J/cm<sup>2</sup>. The untreated control site grew immediately at 8J/cm<sup>2</sup>. We speculate that the other surface damage appearing during the tests was caused by contamination, possibly carbon residue from the dissociation of CF<sub>4</sub>.

Although only a small set of sites were tested, the flame torch shows promise to mitigate growth of heavy laser-initiated damage. Contamination by carbon deposits is also a problem with this method. The hardware is simple and inexpensive, and it could be configured to rapidly process multiple sites on large optics. The method needs additional development, but it could be a reasonable back-up method to the CO<sub>2</sub> process.

## 7. CONCLUSIONS

All of the tested methods, except the plasma torches, gave promising results for mitigating the growth of surface damage sites on polished fused silica. Clearly the CO<sub>2</sub> processing gives the most consistent, convincing results. Moreover, the CO<sub>2</sub> laser processing method should be relatively inexpensive and straightforward to apply, for mitigating sites on large aperture optics. Chemical etching the surface with HF solution also exhibits promise to mitigate laser-initiated damage. However, deep chemical etching would not be acceptable if it aberrates the transmitted wavefront beyond user specifications. Nevertheless, since it is potentially a relatively inexpensive method also, it should be considered as a primary backup to the CO<sub>2</sub> method, for mitigating surface damage on polished silica optics.

One can speculate about any advantage of the flame torch mitigation compared with CO<sub>2</sub> mitigation process. One advantage of the flame torch may be the ability to produce a smoother, shallower pit, which is less disruptive to the optic surface. Also, it may require less capital cost than the CO<sub>2</sub> method. While there may be a cost advantage for implementing the flame torch method, it must be considered as an alternative to the CO<sub>2</sub> process because more tests are needed to determine its reliability. Unless further testing of the CO<sub>2</sub> mitigation process uncovers difficulties, the flame torch approach would not be developed further.

Site-by-site processing of laser damage with a CO<sub>2</sub> laser is feasible and straightforward to apply to large optics (eg., 0.5-1.0m scale). We are developing it as the primary mitigation method for fused silica optics used in high-peak-power applications at 351nm. Furthermore, it is reasonable to envisage a system that does the initiation, identification, and CO<sub>2</sub> processing steps, all at the same time.

## ACKNOWLEDGEMENTS

We wish to acknowledge the help of many LLNL associates who contributed to this effort. Those who performed the growth tests in the slab laser facility; K.Neeb and E.Donohue. Those who provided samples, analysis and interpretation; S.Maricle, L.Sheehan, J.Wong, S.Demos, M.Staggs, J.Ferreira, E.Lindsey and R.Torres. Those who performed the chemical etching; L.Summers and J.Britten, and others who contributed advice; D.Milam, L.Chase, W.Siekhaus and F.Genin.

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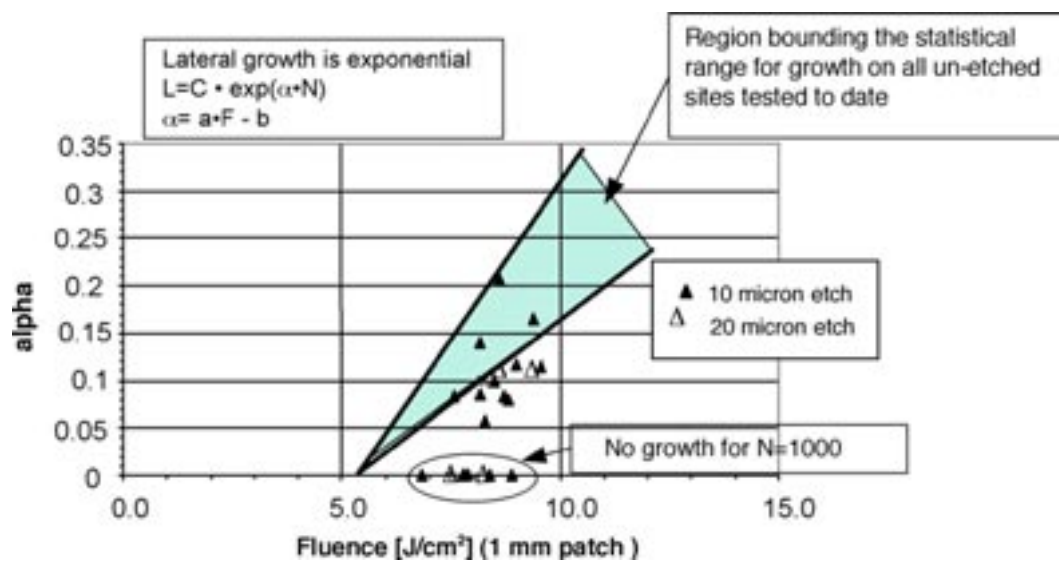


Figure 1. Plot showing results of damage growth experiments for wet chemical etched damage sites relative to the range of data for un-etched sites. About 40% of the etched sites did not grow for 1000 shots at the given fluences.



Figure 2. Photo of a RF micro-torch etching a local site on fused silica.

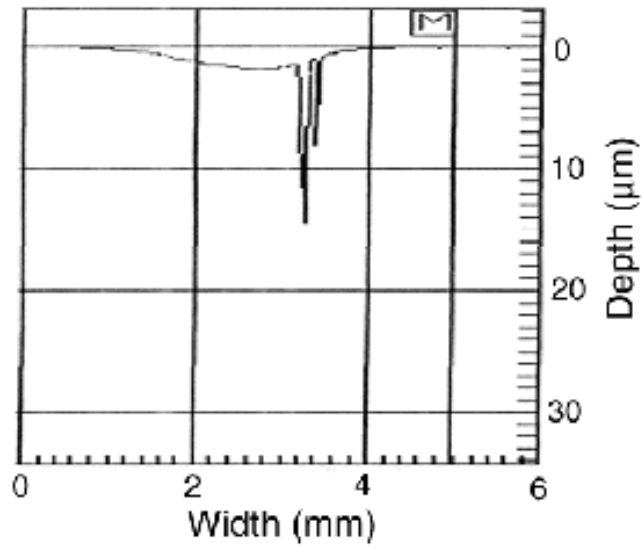


Figure 3. Profile of a 2 $\mu$ m deep pit micro-plasma etched over a 10-15 $\mu$ m deep laser-initiated damage site in fused silica.

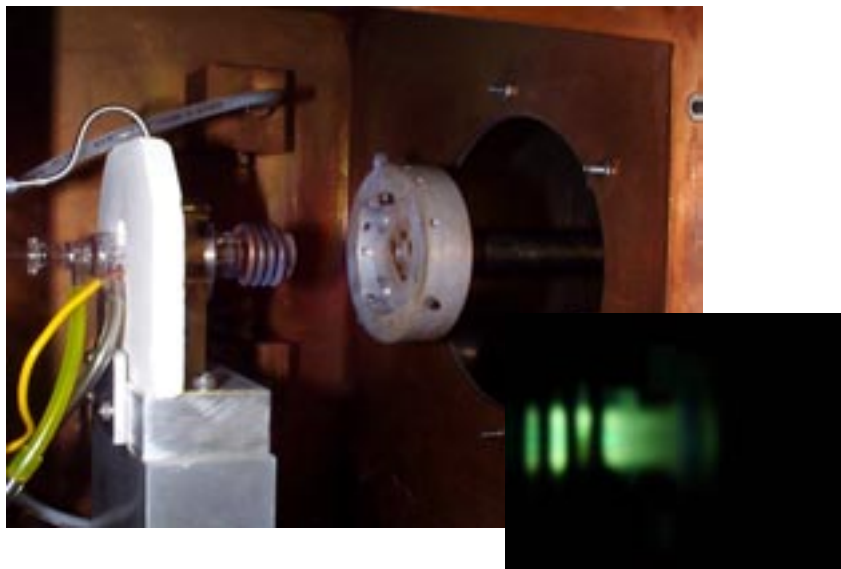


Figure 4. A microwave plasma torch (nitrogen gas) fed by  $\text{CF}_4$  gas, to produced fluorine atoms for reactive atom plasma processing (RAPP).

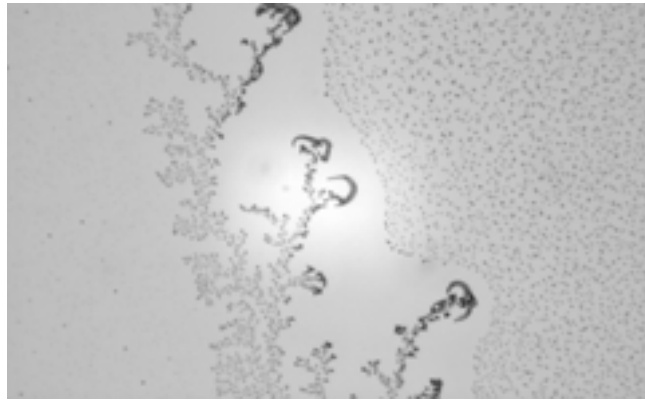


Figure 5. Photomicrograph of carbon containing deposits on the fused silica surface produced by decomposition of carbon-tetrafluoride gas from the plasma torch.

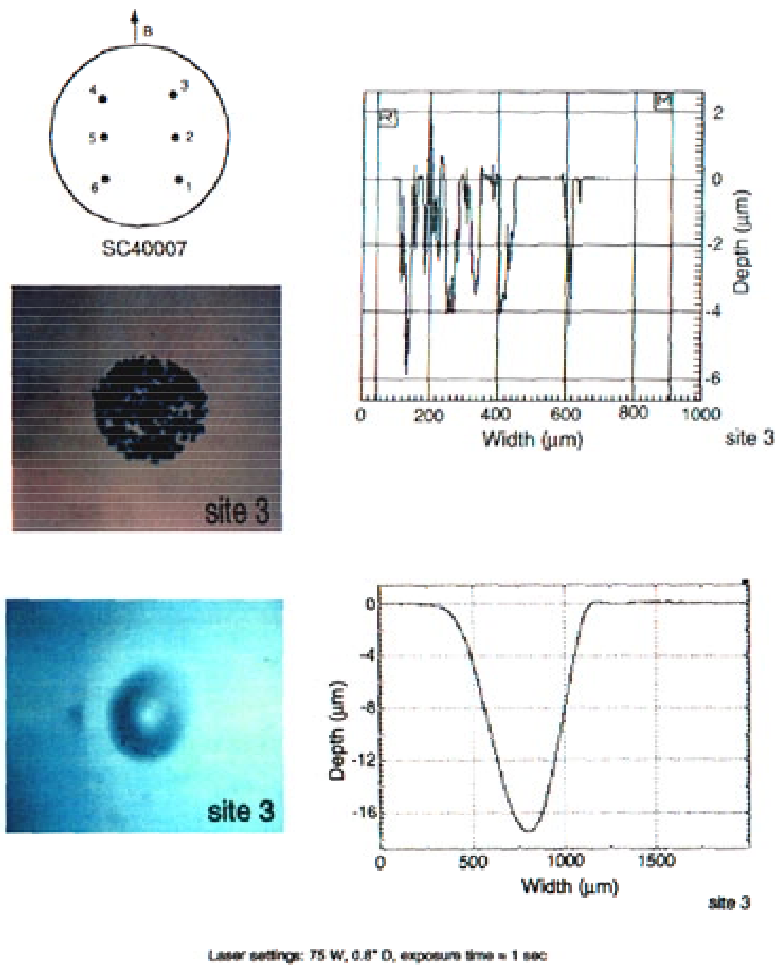


Figure 6. Example of surface damage mitigation by CO<sub>2</sub> laser processing. Many rough pits (top photo and graph) are transformed to a smooth Gaussian-shaped pit (bottom photo and graph) by a single 50W pulse in 1 second.

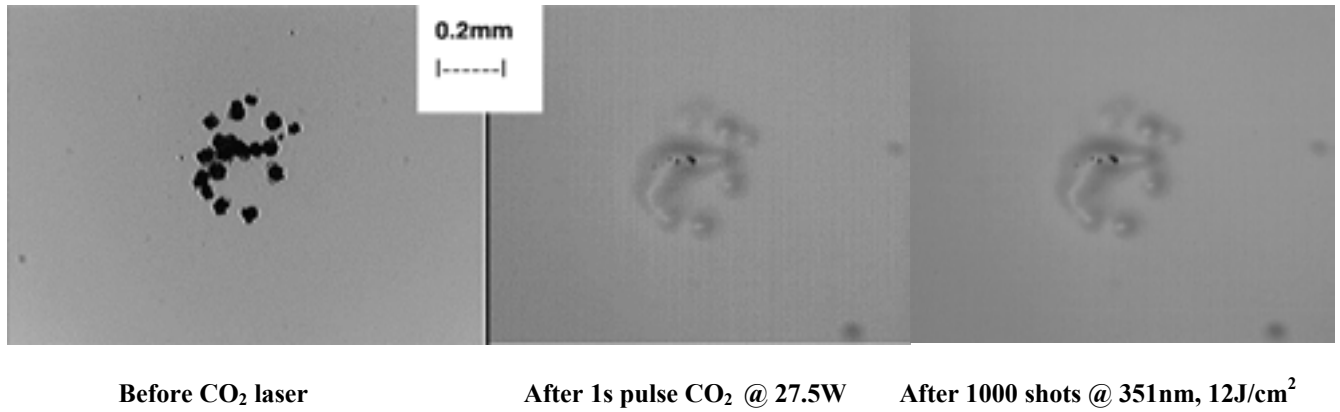


Figure 7. Optical micrographs of a laser-initiated damage site before (left) and after CO<sub>2</sub> processing (middle), and after exposure of 1000 shots at 351nm, 11ns (right), showing that damage growth is completely stopped.

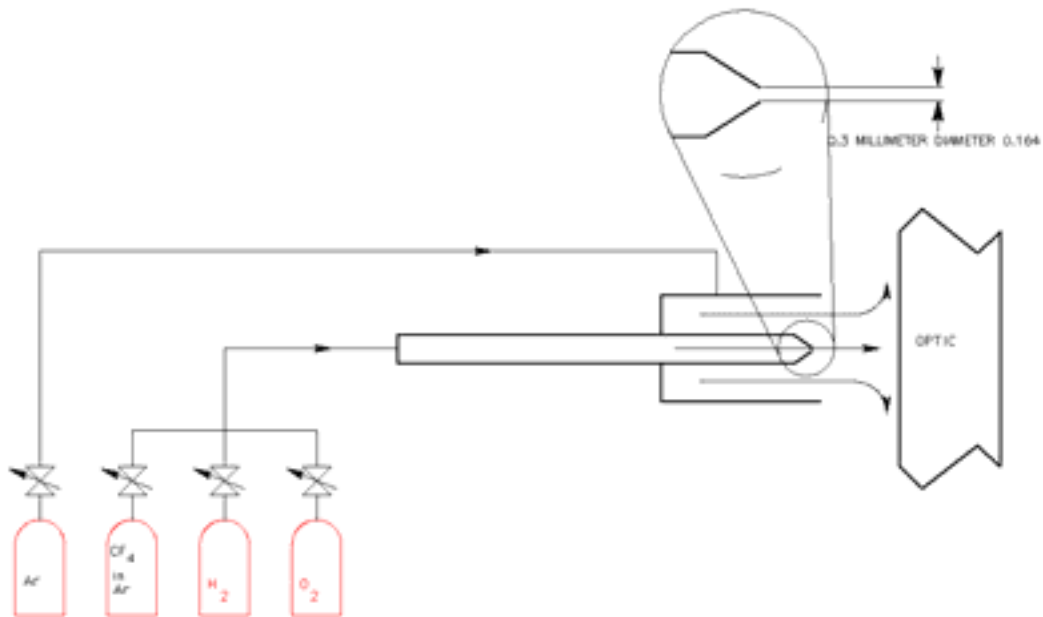


Figure 8. A schematic of the micro-flame torch system.